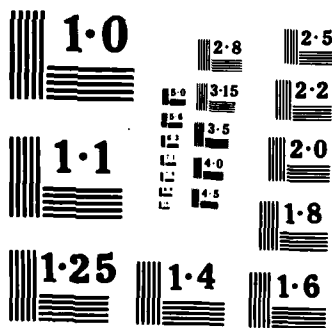END

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
LINCOLN LABORATORY

InP MATERIALS

ANNUAL TECHNICAL SUMMARY REPORT
TO THE
ROME AIR DEVELOPMENT CENTER

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ABSTRACT

This report covers the work on InP materials carried out with the support of the Department of the Air Force during the period 1 October 1983 through 30 September 1984. A part of this support was provided by the Rome Air Development Center. The current objectives of the program are to improve the yield of high-purity polycrystalline InP used as source material for crystal growth and to optimize the liquid-encapsulated Czochralski (LEC) method in order to grow single crystals with low defect density, low residual impurity concentration and uniform dopant concentration.

Electrical measurements were made on samples from 12 of the 15 polycrystalline InP ingots synthesized. Only two had mobilities exceeding 5×10^4 $cm^2V^{-1}s^{-1}$. The decrease in the yield of high-purity ingots compared to previous years can be attributed to the continuing low purity of as-received P and a reduction in the effectiveness of P prebaking. Since secondary-ion mass spectrometric analysis showed that Si is the dominant donor impurity in the less pure ingots, it is probable that as-received P is contaminated with Si.

We have initiated development of a technique for synthesis of InP in the crystal puller prior to LEC growth. One boule obtained by using the in situ technique had a mobility of 2.9×10^4 $cm^2V^{-1}s^{-1}$.

To measure the microscopic growth rate of InP crystals, the LEC system has been modified to permit electric current pulses to be passed through the melt and growing crystal for interface demarcation. In one run the

growth rate was found to vary from less than 1 µm s^{-1} to more than 6 µm s^{-1}.

Promising results have been obtained in initial experiments on the use of a two-piece susceptor to reduce the dislocation density in LEC crystals without lowering the yield of twin-free boules.

INTRODUCTION

The goals of the InP materials program at Lincoln Laboratory are the development of reliable techniques for preparing high-quality InP crystals with controlled electrical properties and the utilization of these techniques to produce crystals needed for research on optoelectronic devices such as InP photoconductive switches and GaInAsP/InP diode lasers and detectors and on electronic devices such as JFETs.

Our program consists of two parts: synthesis of InP from the elements, and crystal growth by the liquid-encapsulated Czochralski (LEC) method. Synthesis is carried out in order to assure an adequate supply of charge material with the purity desired for LEC growth, since such material is not consistently available from commercial sources.

In previous years all synthesis runs were carried out in horizontal sealed silica ampoules by directional solidification from In-rich melts, which yielded polycrystalline ingots for transfer to our high-pressure crystal puller. This year we have initiated development of a technique for synthesis of InP in the puller prior to LEC growth. One boule obtained by using the _in situ_ technique had a mobility of 2.9×10^{4} $cm^{2}V^{-1}s^{-1}$ at 77 K.

In FY84 the yield of polycrystalline ingots exhibiting 77 K mobility values greater than 5×10^{4} $cm^{2}V^{-1}s^{-1}$ was only 2/12, compared with 7/20 in FY83 and 23/44 in FY80-82. Secondary-ion mass spectrometric (SIMS) analysis of ingots with donor densities greater than about 1×10^{16} cm^{-3} showed that Si is the major residual impurity in such ingots. This finding, combined with the results of electrical measurements on many

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ingots, indicates that as-received P is generally contaminated with significant quantities of Si or perhaps with some other impurity that influences the incorporation of Si. For the two best ingots, the degradation in electrical properties resulting from Si incorporation has been reduced by vacuum baking the P at a temperature just below the point at which it sublimes, but P prebaking was ineffective for the other ingots.

In order to measure the microscopic growth rate of InP crystals, we have modified the LEC system so that electric current pulses can be passed through the melt and growing crystal to delineate the crystal-melt interface at regular time intervals. Results for one run show that the microscopic growth rate varied from less than 1 μm s^{-1} to greater than 6 μm s^{-1}.

With the objective of reducing the dislocation density in LEC crystals, we have fabricated a two-piece graphite RF susceptor designed to permit the temperature gradient in the growing crystal to be decreased without lowering the yield of twin-free boules. Several experiments have been carried out with this susceptor. One LEC boule 32 mm in diameter had a very low dislocation density.

SYNTHESIS OF POLYCRYSTALLINE INGOTS

Synthesis of InP from the elements was carried out in horizontal fused silica boats sealed inside fused silica ampoules as described in the report for FY81. Fifteen polycrystalline ingots, each weighing about 1 kg, were synthesized during FY84. As in previous years, Hall coefficient (R_H) and

resistivity (ρ) measurements were made when the grain size was large enough to permit rectangular single-crystal bars at least 1 x 2 x 10 mm^3 to be cut out. All of the samples were n type. The electron concentration at 300 K (n_{300}) was calculated from $n = -e^{-1}R_H^{-1}$, and the Hall mobility at 77 K (μ_{77}) from $\mu = R_H\rho^{-1}$.

Table I lists in chronological order each of the 12 ingots synthesized in FY84 for which electrical measurements could be made, together with the lots of In and P used, the synthesis parameters, and the values of n_{300} and μ_{77} for a sample cut from near the first-to-freeze end of the ingot. The distributions of the μ_{77} values measured for such samples from FY80 through FY84 are shown in histogram form in Fig. 1. From this figure it is evident that the reduction in the yield of high-purity ingots noted in last year's report became even more severe during FY84. Thus the fraction of ingots with $\mu_{77} \geq 5 \times 10^4$ cm^{2}V^{-1}s^{-1} was only 2/12 in FY84, compared with 7/20 in FY83 and 23/44 for FY80 through FY82. Furthermore, for all the FY84 ingots and most of the FY83 ingots the P was vacuum baked in the synthesis ampoule before InP synthesis, while P prebaking was not used for any of the FY80-82 ingots.

In last year's report we suggested that the reduction in the yield of high-purity ingots resulted from a decrease in the purity of the P used as starting material. We attribute the further reduction in yield during FY84 to a combination of two factors: the continuing low purity of as-received P relative to the purity of FY80-82 material and a decrease in the effectiveness with which electrically active impurities were removed (or

TABLE I

SYNTHESIS PARAMETERS AND ELECTRICAL PROPERTIES OF POLYCRYSTALLINE InP INGOTS PREPARED IN FY84

| Ingot | Indium | | | Phosphorus | | | Ampoule design | InP | |
	Source, lot	Vac. bake (°C, h)	Max. boat temp. (°C)	Source, lot	Vac. bake (°C, h)	Synthesis temp. (°C)		n (10^{15} cm^{-3})	μ (10^4 cm^{2}V^{-1}s^{-1})
1	ICA, A1236	900, 6	1060	MCP, 83/01	250, 6	460	1	33	1.2
2	ICA, A1236	900, 6	1060	MCP, 83/01	250, 6	460	1	19	1.8
3	ICA, A1236	900, 6	1060	RASA, 20230	300, 3 200, 4 100, 100	460	2	0.4	9.1
4	ICA, A1236	900, 6	1060	MCP, 83/01	300, 5 200, 72	460	2	1.5	6.4
5	ICA, A1236	900, 6	1060	MCP, 8305	300, 5	460	1	19	1.4
6	ICA, A1236	900, 6	1060	MCP, 8305	300, 5	460	1	19	1.5
7	ICA, A118	900, 6	1060	MCP, 7403	200, 6	460	2	6.4	3.0
8	ICA, JK595	900, 6	1060	MCP, 7403	235, 2 180, 24	460	1	3.8	2.5
9	ICA, JK595	900, 6	1060	MCP, 8307	140, 6	460	1	11	2.0
10	MCP, HR312	900, 6	1060	MCP, 8307	230, 16	460	2	16	1.6
11	MCP, HR312	900, 6	1065	MCP, 7401	170, 7	467	2	16	1.4
12	MCP, HR314	900, 6	1068	MCP, 7605	170, 6	467	2	4.3	3.1

Material Source: MCP - MCP Ltd.
ICA - Indium Corporation of America
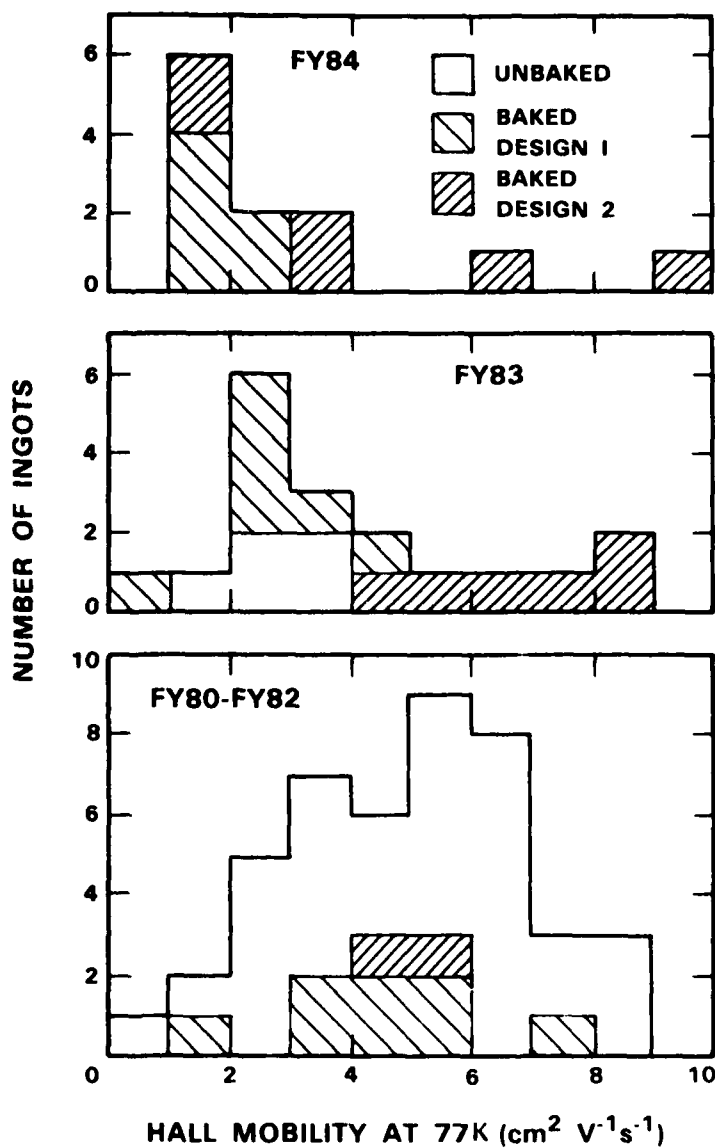RASA - RASA Industries

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Fig. 1. Distribution of electron Hall mobilities at 77 K for polycrystalline InP ingots.

rendered inactive) by P prebaking. In last year's report we noted a correlation between ingot purity and the design of the synthesis ampoules, which differed in the location of the pumping port used during vacuum prebaking of the P. Higher purity was obtained with ampoules in which this port was located between the P reservoir and the In boat than with ampoules in which the port was located at the far side of the In boat from the P reservoir. We suggested that the difference in ingot purity resulted because pumping would be less effective in removing volatile impurities from the P for ampoules with the latter design. For half the ingots synthesized and measured during FY84 we used ampoules with this less effective design, which we have designated "design 1" in Table I and Fig. 1. Consistent with the FY83 results, these ingots had low values of μ_{77}, ranging from 1.2 to 2.5 x 10^4 $cm^2V^{-1}s^{-1}$. For the remaining ingots we used ampoules in which the pumping port was attached directly to the P reservoir. With this design, which is designated "design 2" in Table I and Fig. 1, pumping during the P prebake should be even more effective than with the better of the two FY83 designs, and it was expected that high-purity ingots would be obtained. This was indeed the case for ingots 84-3 and 84-4, which had μ_{77} values of 9.1 and 6.4 x 10^4 $cm^2V^{-1}s^{-1}$, respectively, and n_{300} values of 4 x 10^{14} and 1.5 x 10^{15} cm^{-3}. (The μ_{77} value for ingot 84-3 is the highest we have ever obtained for a polycrystalline ingot, and is particularly noteworthy because ingot 83-2, which was synthesized from the same lot of P but without prebaking, had μ_{77} of only 1.1 x 10^4 $cm^2V^{-1}s^{-1}$ and n_{300} of 2.6 x 10^{16} cm^{-3}.) However, the

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four ingots subsequently synthesized in ampoules with design 2 had μ_{77} values ranging from only 1.4 to 3.1 x 10^4 $cm^2 V^{-1} s^{-1}$, with n_{300} values between 4.3 x 10^{15} and 1.6 x 10^{16} cm^{-3}. Apparently the P used, which came from four different lots, differed from the earlier material in such a way that the effectiveness of prebaking was substantially decreased.

It was noted in last year's report that photoluminescence measurements made in high magnetic fields showed S to be the principal donor impurity in our InP ingots with n_{300} of about 1 x 10^{15} cm^{-3}. Unfortunately, the photoluminescence technique is not applicable to samples with n_{300} greater than about 5 x 10^{15} cm^{-3}. In order to identify the donor impurity or impurities present in ingots with higher values of n_{300}, six samples with $n_{300} > 1$ x 10^{16} cm^{-3} (together with three control samples with $n_{300} < 1$ 10^{15} cm^{-3}) were submitted to Charles Evans and Associates for determination of Si, Ge, Sn, S, Se and Te by SIMS analysis using Cs beam excitation. The results obtained for impurities other than Si are not significant, since the concentrations found for these impurities were essentially at the respective background limits of the SIMS instrument. These limits were less than 10^{15} cm^{-3} for Ge, Se and Te, 1-6 x 10^{15} cm^{-3} for S, and 1-3 x 10^{16} cm^{-3} for Sn. The results of the Si determinations are given in Table II, where the samples are listed in order of increasing n_{300}. The data for the three purest samples establish the instrumental background limit as 1-2 x 10^{15} cm^{-3}. The Si concentrations for the other samples are at least five times higher than this background limit. These concentrations, which are expected to be accurate to a factor of 2 or 3,

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decreases the heat flow outward from the portion of the crystal just above the B_2O_3. As a result there should be a marked reduction in the radial temperature gradients in this critical region and a consequent reduction in dislocation formation. The gap between the upper and lower sections of the susceptor, which is located opposite the B_2O_3, permits heat flow outward from the B_2O_3 and from the portion of the crystal below the surface of the B_2O_3, particularly if a transparent fused-silica crucible is used. This heat flow should produce an increase in the axial gradient in the vicinity of the crystal-melt interface, which should decrease the probability of twinning. The details of the heat flow pattern can be adjusted by varying the design of the upper section of the susceptor, the width of the gap between the two sections, the apportionment of the RF coils between the two sections, and the height of the B_2O_3.

Several growth runs have been made using a two-piece susceptor with the configuration shown in Fig. 4. In these runs we employed a fused-silica crucible, a 40-mm height of B_2O_3, and a (111) seed with its B surface facing the melt. The most recent run yielded a 32-mm-diameter boule that was untwinned for more than half its length. For a (111) slice cut about 30 mm below the seed, over most of its area the density of pits produced by a dislocation etch was less than 100 cm^{-2}. On the basis of this encouraging result we are planning additional runs with the two-piece susceptor in order to determine the optimum growth procedure for obtaining both low dislocation densities and a low incidence of twinning.
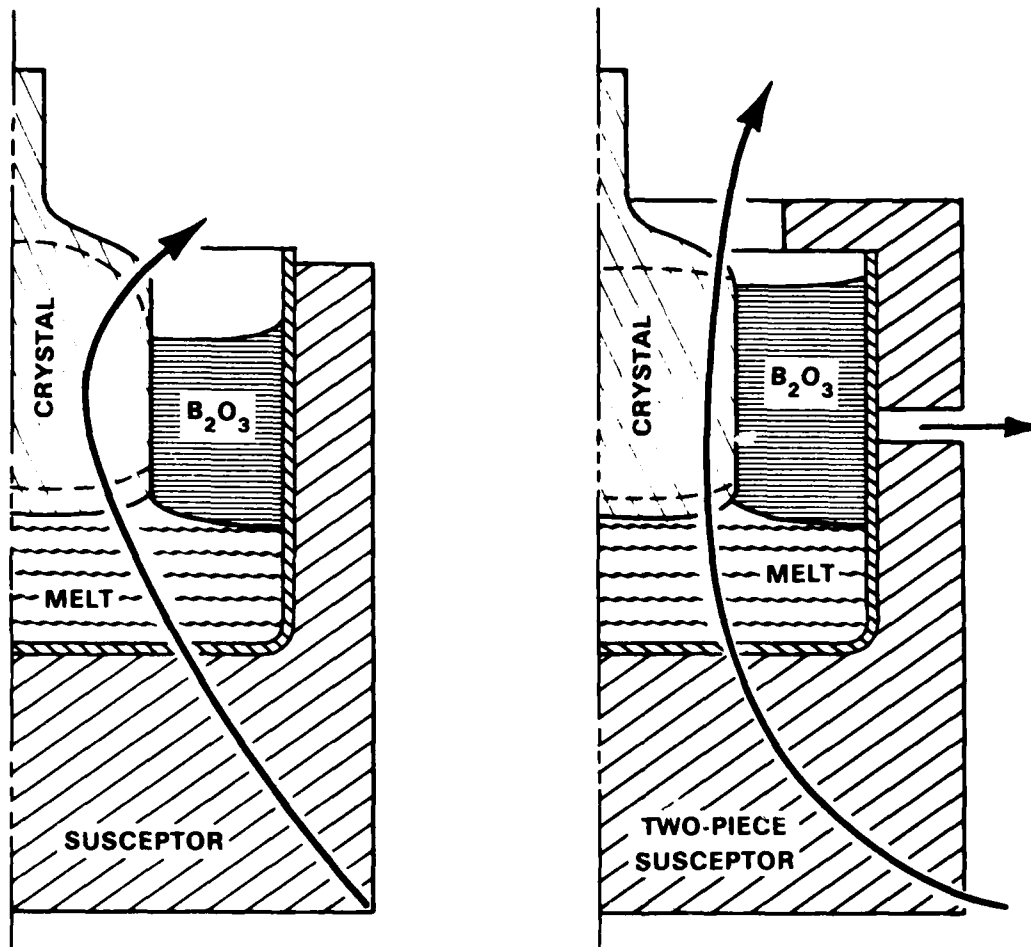
Fig. 4. Diagrams showing longitudinal cross section through InP crystal
and center section of LEC crystal growth assembly. Solid curves with
arrows indicate heat flow lines, and dashed curves represent temperature
isotherms inside the crystal. Left: assembly with standard one-piece
susceptor. Right: assembly with two-piece susceptor.

several runs with a two-piece susceptor. The configuration of this

susceptor is depicted by the diagram at the right side of Fig. 4, which

shows a longitudinal cross section through a growing crystal and the center

section of the crystal growth assembly, drawn approximately to scale. The

diagram at the left side of Fig. 4 shows a similar diagram for our standard

assembly with a one-piece susceptor. In each diagram the expected heat

flow lines are indicated schematically by the solid curves with arrows,

while the dashed curves represent temperature isotherms inside the

crystals.

The heat flow pattern shown in the diagram for the one-piece susceptor

has been established by Muller, Vokl and Tomzig (5), who made temperature

measurements with thermocouples embedded inside growing InP crystals.

Because the B_2O_3 encapsulant is a very poor thermal conductor compared to

molten InP and high-pressure Ar gas, in the portion of the crystal that is

well below the surface of the B_2O_3 the heat flow is predominantly upward,

and the radial temperature gradients are relatively small. For the portion

of the crystal in the vicinity of the B_2O_3 surface, cooling by the Ar gas

results in substantial outward heat flow, and there are large radial

gradients. Most of the dislocations in the crystal are presumably formed

in this region during the time that the temperature is still high enough

for crystallographic slip to be produced by the thermal stresses generated

by the radial gradients.

The Upper section of the two-piece susceptor, which covers the top

edge of the crucible and extends inward, serves as an after-heater that

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The use of the graphite crucible rather than the usual PBN crucible during the measurement runs almost certainly caused a major perturbation in the growth conditions. To permit the PBN crucible to be used, we have designed a small graphite electrode that can be inserted into the melt to provide a path for the current. Experiments using this electrode are planned for the near future.

LEC GROWTH WITH A TWO-PIECE SUSCEPTOR

Crystals of InP grown by the LEC method generally contain high densities of dislocations, which are formed because a region still at high temperature after solidification is subject to radial temperature gradients that generate thermal stresses sufficient to induce crystallographic slip. Dislocation formation is particularly severe for InP because its values of thermal coductivity and critical resolved shear stress are relatively low (compared to those of Si, for example). In experiments on LEC growth reported previously (4), we achieved significant reductions in the dislocation density of InP crystals by decreasing the temperature gradients during growth. However, decreasing the gradients also increased the probability of twinning, which became unacceptably high well before they could be decreased sufficiently to eliminate the dislocations.

In all previous LEC growth runs we used a one-piece graphite susceptor for heating the crucible and InP melt. With the objective of modifying the temperature gradients in order to further reduce the dislocation density while maintaining a high yield of twin-free crystals, we have recently made
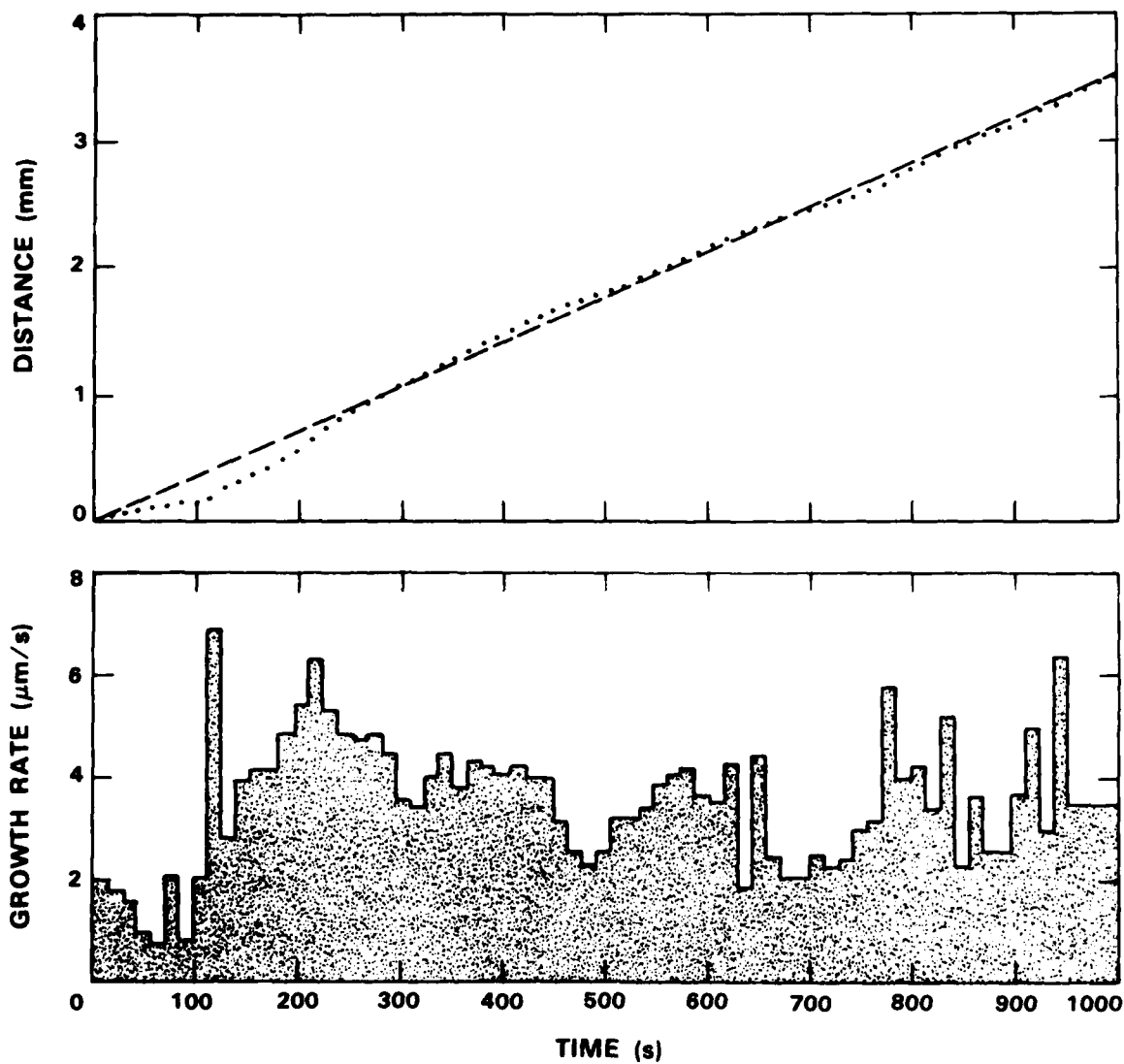
Fig. 3. Time dependence of data obtained by electric current pulse technique for interface demarcation. Above: distance between initial striation and each later striation. Below: average microscopic growth rate between successive pairs of pulses.

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doped with Sn to about 10^{18} cm^{-3}. For the two runs we replaced the usual PBN crucible with a graphite crucible and installed an insulated Ni rod that ran up the center of the crucible support shaft from below the pressure chamber until it made contact with the graphite susceptor. A simple capacitor discharge circuit was employed to pass positive current pulses of 200-300 A from the seed shaft through the seed (which was soldered to a stainless steel holder) and then through the melt, the crucible, the susceptor, and finally the Ni rod. These pulses were large enough to produce striations that could be observed by Nomarski interference microscopy after a hot ferric chloride etch.

The results for the two runs were very similar. The data for one run, in which the interval between current pulses was 14 s, the seed rotation rate was 40 rpm, and the crucible was not rotated, are shown in Fig. 3. The upper part of the figure is a plot showing the total vertical distance from the striation produced by the first pulse (which was located somewhat below the point of seed attachment) down to the striation produced by each of the following pulses. The lower part is a bar graph showing the average growth rate between each pair of successive pulses. This average rate varies from less than 1 to more than 6 μm/s. The magnitude of the deviation between the experimental points and the straight line in the upper plot exhibits a variation of over 0.5 mm. Since the pull rate for the seed shaft has been found to be constant, this variation shows that the height of the liquid column from the bottom of the boule to the free surface of the InP melt varied by the same amount during the run.

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a large fraction of the P was injected into the melt before the run was terminated. It appears that the major problems have now been solved, and we expect to achieve a good yield of 600-g single-crystal boules in the near future. A recent run produced a boule consisting of several large grains, one of which yielded a sample with a μ_{77} value of 2.9×10^4 $cm^2V^{-1}s^{-1}$, compared with 4.2×10^4 $cm^2V^{-1}s^{-1}$ for our best LEC boule grown from a stoichiometric charge synthesized outside the puller.

MICROSCOPIC GROWTH RATE OF LEC CRYSTALS

As described in the report for FY83, we have found that the homogeneity of the dopant distribution in LEC InP crystals can be greatly improved by increasing the rate of seed or crucible rotation. Since the incorporation of impurities from the melt into a crystal depends on the microscopic growth rate, it is of interest to measure this rate as a function of the growth parameters, particularly the rotation rates. In order to make growth rate measurements we have modified the LEC system to permit the use of the interface demarcation technique demonstrated by Gatos, et al. (3) for Ge and InSb. This technique employs electric current pulses to momentarily change the microscopic growth rate and therefore the impurity concentration. These pulses are applied at regular intervals to produce impurity striations whose spacing gives the average microscopic growth rate in the interval between successive pulses.

We have used the interface demarcation technique to measure the microscopic growth rate during the growth of two 2.5-cm-diameter boules

appear at the melt-B_2O_3 interface. If the reservoir is heated at a suitable rate, most of the bubbles disappear as the P vapor dissolves into the In melt, while the rest rise to the surface of the B_2O_3, where they burst to release Ar. If the reservoir temperature is raised too rapidly, the rate of bubble formation is so high that the bursting bubbles release P as well as Ar, causing the puller windows to become coated with an opaque film of P. If the average heating rate is too low, temperature fluctuations in the puller can produce a momentary decrease in reservoir temperature, and the resulting decrease in reservoir pressure can permit liquid from the crucible to rise far enough into the injector tube to solidify, sealing off the tube and aborting the run. To avoid these extremes, the heating rate is adjusted to maintain a satisfactory rate of bubble formation. When the reservoir temperature reaches 580°C it is held constant for the remainder of the run. (From the vapor pressure of P at this temperature, which is slightly higher than the reported vapor pressure in equilibrium with molten InP at its melting point, it is estimated that the reservoir contains about 10 g of P in the vapor phase.) After the bubbling stops, indicating that the melt has reached essentially stoichiometric composition, the InP seed is lowered into the melt and crystal growth is performed in the usual manner.

Twelve runs employing in situ synthesis have been performed to date. For a variety of reasons (including insufficient reservoir heating rate, reservoir heater burnout, clouding of the B_2O_3, and coating of the puller windows) most of these were unsuccessful, but even in the unsuccessful runs

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The top of the P reservoir is initially attached to an open ended silica pumping tube. The reservoir is loaded by dropping small P chunks through this tube, which is then o-ring sealed to an ion-pumped vacuum system. The injector tube is closed off at the bottom with an o-ring-sealed cap, the P is vacuum baked at a temperature just below the point at which it begins to sublime (usually 200-250°C), and the reservoir is sealed by pulling off the pumping tube to form the point shown at the top of the reservoir in Fig. 2. The In metal is generally loaded into the LEC crucible without B_2O_3 and vacuum baked at 800-900°C in the crystal puller.

Next the puller is opened, a B_2O_3 cylinder is placed over the In in the crucible, the cap is removed from the reservoir injector tube, the reservoir assembly is installed in the position shown in Fig. 2, and the puller is closed and charged with Ar gas at atmospheric pressure. The RF power is turned on, and the In is heated to a temperature just above 1065°, the melting point of InP. The Ar pressure is then raised to about 600 psi and the P reservoir heating cycle begun. When the thermocouple in the reservoir reaches about 300°C, the reservoir assembly is lowered (by lowering the seed shaft) until the bottom of the injector tube is near the bottom of the In melt. The assembly remains in this position for the remainder of the run. As the temperature of the reservoir is further increased, the P vapor pressure increases; when the total pressure in the reservoir (the sum of the Ar and P partial pressures) reaches about 0.1 psi above the Ar pressure in the pressure vessel, gas bubbles are observed to
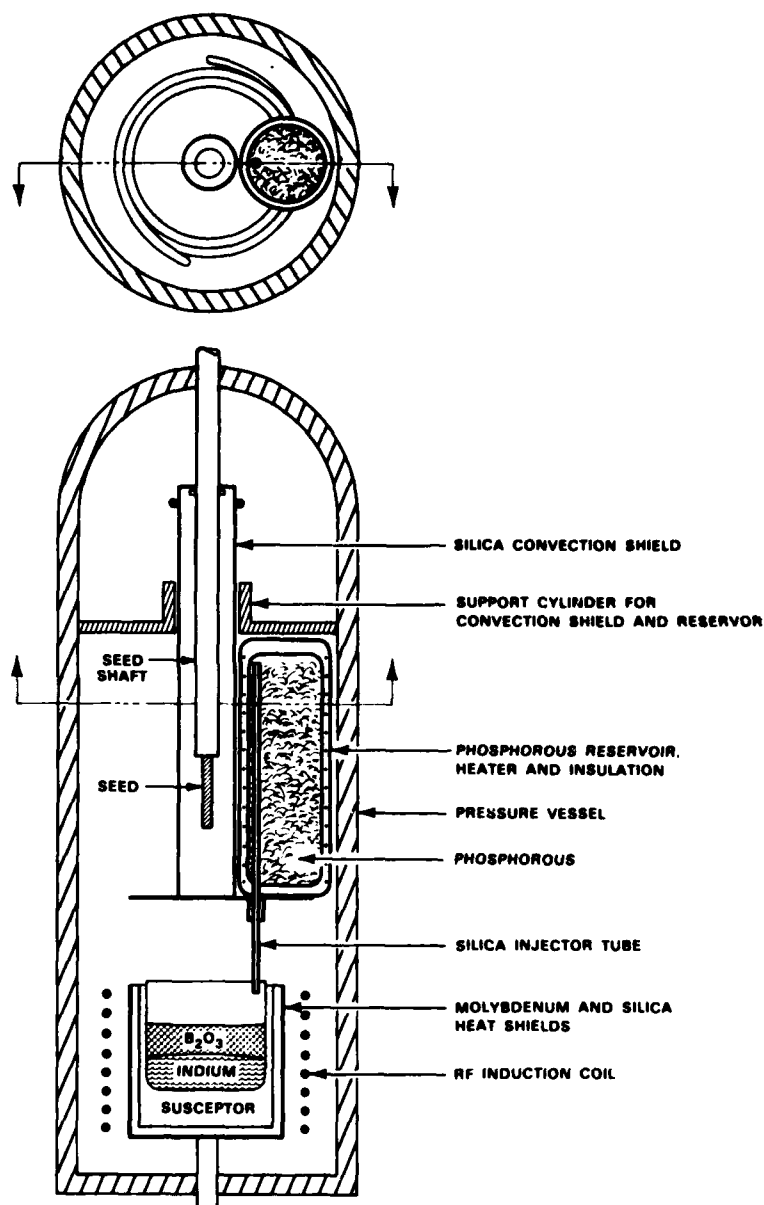
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Fig. 2. Apparatus for _in situ_ synthesis and LEC growth of InP.

several workers have performed in situ synthesis by placing the P in a separate container inside the pressure vessel and heating this container to inject the P via the vapor phase into an In melt encapsulated with B_2O_3 (1,2).

With the objective of using the injection technique to prepare 500-600 g melts of InP in our LEC puller we have designed and constructed the apparatus shown in Fig. 2. This figure is a sketch, drawn approximately to scale, of vertical and horizontal cross sections through the loaded crystal puller just before beginning synthesis. The RF heated susceptor shown near the bottom of the sketch holds a fused silica or PBN crucible that contains the In and B_2O_3. Above the susceptor on the right side is a fused silica P reservoir, which can hold about 130 g of P. A small reentrant tube (not shown in the figure) is sealed into the P reservoir as a thermocouple well. An injector tube open at both ends is fused into the bottom of the reservoir and extends below the bottom for a distance approximately equal to the crucible height. The reservoir is wrapped with resistance heater wire and zirconia insulating cloth, then placed inside an outer silica vessel that holds the cloth in place and provides more thermal insulation. The whole reservoir assembly is attached to a transparent silica convection shield, which is constructed with a precision ground tube that can slide inside a stainless steel support cylinder attached to the pressure vessel near the top. At the outset of a growth run a pin in the seed shaft holds the convection shield and reservoir at the position shown in Fig. 2.

fact that P prebaking is sometimes very effective in improving the electrical properties of the ingots. At this time we cannot identify the hypothetical volatile Si species, although we speculate that it might be silane or some other silicon hydride. In addition, we cannot eliminate the possibility that the Si found in the low-purity ingots actually comes from a source other than the P. In that case there would have to be some mechanism by which impurities in the P influence the degree to which Si in electrically active form is incorporated into the InP during synthesis.

IN SITU SYNTHESIS AND LEC GROWTH

The in situ synthesis of GaAs in LEC pullers prior to crystal growth is a well-established commercial process. Compared to synthesis in a separate system, in situ synthesis achieves higher crystal purity because handling is reduced and also yields savings in time, equipment and container material. For the in situ process, elemental Ga and As are loaded into the LEC melt crucible under a tight fitting B_2O_3 pellet, and reaction between the elements takes place as the temperature is raised, with only small As losses.

The potential advantages of in situ synthesis are the same for InP as for GaAs. Because of the high vapor pressure of P, however, in situ synthesis of InP cannot be accomplished simply by heating a mixture of In a P under B_2O_3 in the melt crucible, since LEC pullers are not designed to withstand the pressures that would be required to contain the encapsulated in the crucible until reaction is complete. To overcome this difficulty

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are approximately equal to n_{300} for ingots 83-15 and 83-22, about 3 x n_{300} for ingots 84-4 and 84-20, and about 4 x n_{300} for the other two ingots. It is clear that Si, not S, is the major donor impurity in these relatively impure ingots. In addition, the data suggest that in some cases a significant fraction of the Si is not electrically active. The presence of Si in our early ingots of comparable purity was established by a spark source mass spectrographic investigation conducted in FY79, which showed that the Si concentrations were roughly correlated with the values of n_{300} in the range of 2 x 10^{16} to 2 x 10^{17} cm^{-3}.

In the previous discussion of the electrical properties of InP ingots synthesized under different conditions from different lots of In and P, we attributed the low purity of many ingots prepared during FY83 and FY84 to the presence of electrically active impurities that originated in the as-received P and were not removed (or rendered inactive) by P prebaking. If this model is correct, from the SIMS results for such low-purity ingots it would follow that the dominant impurity species in the P is Si. According to the model, since the synthesis process involves the vapor transport of P from its original location in the ampoule to the boat containing molten In, incorporation of Si in the ingots would require that Si be transported through the vapor phase along with the P. However, elemental Si is far less volatile than P. The model therefore implies either that a volatile Si-bearing species is initially present in the P or that such a species is formed during the synthesis process. The existence of a Si-bearing species even more volatile than P would account for the

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TABLE II

CONCENTRATIONS OF Si FOUND BY SIMS ANALYSIS

	In	P	[Si]	n_{300}	
Ingot	Source, lot	Source, lot	$(10^{16}\ cm^{-3})$	$(10^{16}\ cm^{-3})$	$[Si]/n_{300}$
83-5	ICA, A117	MCP, 8201	(0.1)*	0.011	---
83-4	ICA, A118	MCP, 8201	(0.2)*	0.058	---
83-16	MCP, HR/136	MCP, 8201	(0.2)*	0.14	---
83-15	MCP, HR/136	MCP, 8201	1	0.95	1
84-10	MCP, HR/312	MCP, 8307	4	1.6	2.5
83-2	ICA, A118	RASA, 20320	10	2.6	4
84-1	ICA, A1236	MCP, 83/01	10	3.3	3
83-22	MCP, HR196	RASA, 30424	4	4.2	1
82-WW	MCP, HR/134	RASA, 20320	70	16	4

*Instrumental background

Material Sources:
MCP - MCP Ltd.
ICA - Indium Corporation of America
RASA - RASA Industries

ACKNOWLEDGMENTS

REFERENCES

1.   S. B. Hyder and C. J. Holloway, Jr., J. Electron. Mater. 12, 575 (1983

2.   J. P. Farges, J. Crystal Growth 59, 665 (1982).

3.   H. C. Gatos, A. F. Witt, M. Lichtensteiger, and C. J. Herman, NASA Spec. Publ. SP, 77 (NASA SP-412, Apollo-Soyuz Test Proj. Summ. Sci. Rep. vol. 1; N78-17088) 429-447.

4.   G. W. Iseler, J. Electron. Mater. (to be published).

5.   G. Muller, J. Vokl, and E. Tomzig, J. Crystal Growth 64, 40 (1983).

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This report covers the work on InP materials carried out with support of the Department of the Air Force during the period 1 October 1983 through 30 September 1984. A part of this support was provided by the Rome Air Development Center. The principal donor in less pure polycrystalline ingots was identified as Si, which probably originated in the as-received P. Initial experiments have been performed on *in situ* synthesis of InP in the LEC puller, measurement of the microscopic growth rate of LEC crystals, and the use of a two-piece susceptor to reduce the dislocation density in these crystals.

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